

# NIMS

2008.Vol.6 No.6 June

# NOW

## International

Materials - The Key to  
a Sustainable Society

**FACE** フェイス  
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Rotor for compact power-generating turbines using a new alloy developed by NIMS.

NIMS is actively grappling with the development and practical application of various types of ultra-high temperature materials and new coating materials, beginning with high performance Ni-based superalloys. Realization of advanced jet engines and high efficiency power-generating turbines will significantly reduce fuel consumption and CO<sub>2</sub> emissions. Ultra-high temperature materials with heat resistance at higher service temperatures make this possible.



# Materials - The Key to a Sustainable Society

Today, innovation which contributes to preserving the global environment while ensuring energy and resource security is urgently required. As a result, even higher expectations than in the past are now placed on materials. How can NIMS respond to these needs? We are exploring the vistas of materials research from the viewpoint of energy and the environment.

## ◆◆ Materials are the key to innovation.

Industrial machinery has transformed society, aircraft have brought about a revolution in travel and logistics, and IT has woven the world together in an unprecedented information network. However, we are seldom conscious of the fact that new materials are the “parents” of all of these innovative technologies. Steel and aluminum have given birth to industrial machinery and aircraft, and of course, silicon has made information technology possible. Without these materials and the technologies that create them, humankind would not enjoy these benefits. It is no exaggeration to say that materials technologies have changed history and given birth to a new society.

Looking back on the progress achieved in familiar electrical and electronic products, one comes to the same realization. For example, television technology has progressed from the original cathode ray tube (CRT) to liquid crystal displays and organic EL, allowing users to enjoy more beautiful images with simpler, more trouble-free devices. Likewise, lighting has evolved from the original incandescent light bulb to fluorescent lighting and now light-emitting diodes, providing safe lighting with substantially reduced electric power consumption. As these examples show, materials are also the key to innovation in electronics.

## ◆◆ A materials researcher's scenario for the future.

The first commitment period under the Kyoto Protocol finally began in April of this year. As a short-term target, Japan must reduce its emissions of greenhouse gases (GHG) by 6% in comparison with the baseline year of 1990 during the 5 year period beginning in 2008. As Japan prepares to host the Lake Toya Summit in Hokkaido this summer, a more ambitious long-term goal of reducing GHG emissions by 60-80% by 2050 has emerged.

Meanwhile, the prices of energy and metal resources have risen dramatically since the beginning of 2008, and difficult conditions are continuing, in which nobody can ignore the urgency of effective utilization and recycling of resources. In other words, a fundamental response to energy, resource, and environmental problems is an issue which materials research must address without delay.

In recent years, Japan has continued to develop innovative energy saving technologies which improve energy efficiency in manufacturing, and is now the world's leader in energy efficiency (Fig. 1). However, with diffusion of these technologies already at substantially 100%, there is little room for further improvement along these lines, and innovations which consider the environment will be necessary when aiming at even higher efficiency. Given this situation, there are rising calls for materials which can produce innovation from the most upstream processes with the aim of realizing a sustainable society.

What scenario do materials researchers present for this future? Dr. Kotobu Nagai, Coordinator of the Materials Research for the Environment and Energy area at NIMS, mentions the following three points as a fundamental stance which opens the way for contributing to solutions to resource problems and energy problems with innovative materials technologies, and as a result, contributing to a solution to environmental problems.

The first point concerns networks. Dr. Nagai comments, “In order to obtain energy efficiency by making good use of resources, it is necessary to consider the network as a whole, and not simply the individual technologies. An answer as to how materials research should contribute is more readily apparent if the problems are organized based on a consideration

of flows through networks, for example, in power transmission and logistics, including the balance of distributed and centralized systems.”

Distributed energy production includes technologies like solar and wind power generation. However, these technologies have certain inherent problems. In addition to large fluctuations in generating capacity, power transmission efficiency is not particularly high. For these reasons, systems for storing power are indispensable for ensuring a stable power of supply in the network as a whole. A good balance with the power supply from large power plants, which are centralized facilities, is also necessary. There should be many situations in which materials can play an active role, in both power generation and power storage.

Dr. Nagai's second point is the necessity of an international perspective: “The European countries are extremely active in wind power, which is frequently mentioned in Japan as well. In actuality, however, in no small number of cases, these countries cover much of their energy demand by importing power generated at nuclear power plants in neighboring countries. Thus, energy production is not simply conceived in single country units; a perspective which encompasses a certain region extending beyond national borders is necessary. The same can also be said of resource and waste problems. In recent years, exports of scrap metal and used cars from Japan have increased dramatically, reaching a scale where one must say that the country is basically ‘hemorrhaging’ resources. We would like to find a mutually beneficial direction for solving this problem through cooperation with nearby importing nations. For this, sharing of technology and the creation of international standards will also be necessary.”

The third point is a challenge which is the unique dream of materials research. “Our aim is not limited simply to measures which reduce CO<sub>2</sub> emissions or purify the environment. Our ultimate goal in materials research is to reduce total emissions and realize a low carbon society by positively utilizing CO<sub>2</sub>, as a more forward-looking approach with a view to the long term. It is precisely this positive kind of conception which will enable science and technology to contribute to humankind. One example is ‘artificial photosynthesis.’” Dr. Nagai also notes that a paradigm change is needed: “In the past, Japan has based its thinking on the idea that the country is ‘poor in natural resources’. However, we need to think about scenarios that begin from a recognition that this is, in fact, as ‘resource rich country’, as seen in the exports of used cars and the vast accumulation of important industrial metals – or ‘urban mines’ – which now exist in used electronic products.” Thinking about reduction of CO<sub>2</sub> emissions and energy saving tends to suggest images of ‘doing without’ and ‘looking backward’, but materials researchers want to think about these issues more positively.

## ◆◆ The enormous need for materials research.

A variety of needs exist in all respects in materials technologies for resources, energy, and the environment. In January of 2008, NIMS sponsored a roundtable on “The Future



Kotobu Nagai

Coordinating Director  
Materials Research for the  
Environment and Energy

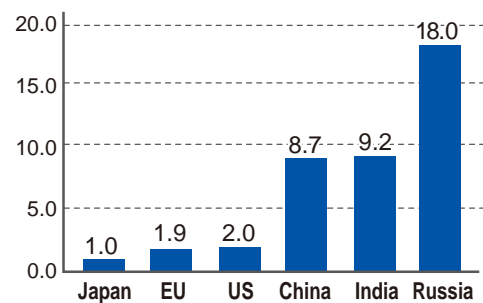
of Materials for the Environment and Energy,” where we received a large number of concrete requests from the assembled researchers, who included scientists from public research centers in Japan and others, as well as persons responsible for public policy. Experts from many fields, including marine science, electric power, transportation and logistics, railways, aerospace, nuclear power, waste, the environment, civil engineering, and others actively exchanged ideas and information from various points of view regarding their expectations for materials research. The valuable opinions exchanged and wealth of recommendations suggested how large and diverse the expectations placed on materials research actually are. Several of these are introduced in the following.

First, in energy production, utilization of natural energy, and particularly solar energy, is the ultimate goal. Although technologies for solar power generation have been developed from an early date, revolutionary materials development is required in order to achieve dramatic increases in generating efficiency and conversion efficiency. The government has set a goal of covering 10% of Japan's total electric power demand with solar power by 2030, but in this, there are numerous issues for materials development, aiming at high efficiency, multi-functionality, long life, and low cost. At present, a wide range of materials are objects of study, including not only polycrystalline silicon and thin film silicon, but also compound thin films, dye sensitized substances, organic materials, and others. There is also a view that flexible transparent inorganic materials are promising candidates for thin film solar cells. Moreover, in addition to the cells themselves, there is a high need for long life, recyclable package materials. Space-based solar power generation, in which solar power is generated in space and beamed back to earth by microwave or laser, is in the experimental stage. Materials are also the focus of active research in many related areas, such as the development of heat-transport media, film-structured mirrors for collecting sunlight, ultra-lightweight structural materials, and the others.

Where automobiles are concerned, catalysts with even higher activity are essential for reduction of exhaust gas pollution, and materials are expected to play a key role in weight reduction and downsizing of automobiles, which are required in order to reduce energy consumption. The future is also

Fig.1

### National comparison of energy consumption by GDP



Ratio of energy consumption (oil conversion-ton)/real GDP, converted to Japan = 1.  
Source: IEA Energy Balances of OECD Countries 2003-2004 (2006)

Japan is the world's most advanced energy saving country; however, further technical innovation is expected.



likely to see hybrid railway rolling stock using battery systems and electric trains powered by fuel cells. The development of electric trains which are capable of operating without overhead power lines will be extremely significant from the viewpoint of resource saving. Here as well, the development of a variety of materials will be required.

The necessity of materials development which is not limited to materials that can achieve their theoretical limits, but makes it possible to raise those theoretical limits, was also strongly emphasized. When the views of the roundtable were collected, it was noted that "Proposals on a large scale, and not simply research focusing on improvement, is expected." Other comments mentioned a desire to "link materials research, from the most upstream creation of new substances to new materials as such, and to link new materials to new products," and stressed that "Research can only be called innovation when it is accompanied by a concept which begins from basic research." Thus, this was an exchange of views where all those present strongly felt that "Materials are key".

◆◆ Proposals that are possible "Because we are NIMS."

Now, let's ask about the actual research topics at NIMS, and how NIMS can respond to diverse needs with new proposals.

One subject that Dr. Nagai mentions as a fascinating topic is "water". Water is an extremely interesting substance, so much so that researchers consider it one of the Seven Wonders of the World. Water takes various complex states, for example, displaying its greatest density at 4°C, forming amorphous ice with the molecular state, and is a mixture of hydrogen ions and hydroxide ions. Because it is weakly diamagnetic, it has the unique property of floating in a strong magnetic field. Is it perhaps possible to use this unique sub-

stance, water, as a material? It would seem possible to develop a new research field based on an innovative understanding of water as a new material. For example, NIMS has proposed a revolutionary water purification method which takes advantage of the diamagnetism of water.

"Soil" is also a topic with considerable potential. At present, steel makers discharge as much as 70 million tons of the byproduct called slag when producing 100 million tons of steel. Based on the novel concept that slag isn't waste, it is in fact an outstanding compound oxide, NIMS is taking on the challenge of utilizing slag. Envisioning increased food production and marine development, NIMS is exploring methods of effective utilization of slag as "soil".

Research and development utilizing nanotechnology also have great potential. NIMS is widely known as a pioneering research center in the field of nanotechnology. In concepts that explore the nano level, for example, molecular recognition of material surfaces, joint work involving materials and chemistry is necessary. Based on nanotechnology as a keyword, innovative technologies which are possible "because we are NIMS" are expected in the fields of energy, resources, and the environment (Fig. 2).

The existence of numerous needs is undoubtedly a great treasure for materials research in connection with energy and the environment. However, there is now a common recognition in all fields that conventional technologies which attempt to improve the performance of existing materials by further development along the same lines are reaching their limits. As Dr. Nagai suggests, in creating new technologies which make it possible to exceed the conventional limit values, a bold change in thinking will be necessary. To achieve this, we will need a conception of networks that does not limit its view to individual fields of industry, and new proposals and approaches for materials based on a long-term vision.



Chikashi Nishimura  
Managing Director, Fuel Cell Materials Center

Encouraged by his success in combustion synthesis of hydrogen absorbing alloys, Dr. Nishimura is now engaged in research on metallic materials related to hydrogen, such as hydrogen separation membranes. Talking enthusiastically about the pleasures of his work in the development of hydrogen materials, he describes dreams with a gaze firmly fixed on the future: "Many of the conventional separation membranes are based on organic materials, but I believe the metal based separation/permeability membranes conceal many unknown possibilities."

## The Fascination of Hydrogen Materials

Laying the foundations for a hydrogen-based society with hydrogen absorption, separation, and power generation

### Have you been involved in materials research related to hydrogen since joining NIMS?

When I completed my Masters degree, I joined one of NIMS's predecessor organizations, the National Research Institute for Metals. My first work there was research and development on oxide dispersion strengthened heat resistant alloys. I did all the fundamentals of powder metallurgy, which included preparation and forming of powders, recrystallization in a single orientation, and so on. This was an extremely valuable learning experience.

My next area of research was combustion synthesis. This is a method of producing substances from powders utilizing the heat of chemical reactions, and is simpler and enables more precise control of the microstructure than the dissolution method. At the time, there was a boom in new materials based on intermetallic compounds, as represented by shape memory alloys and hydrogen absorbing alloys, among others, and I was asked to create a hydrogen absorbing alloy by the combustion synthesis method. Although this was a difficult assignment, after investigating the reaction temperature and reaction heat, I was ultimately successful in experiments with zirconium-nickel. Of course, I was very happy with this result. This was the first time I felt some relationship with hydrogen. Later, I did research on hydrogen embrittlement of structural materials and metallic materials which are characterized by extremely rapid diffusion of hydrogen such as hydrogen separation/permeability membranes.

For me, they're really interesting. If you increase the surface area of a hydrogen absorbing alloy by crushing it, and then supply hydrogen at 1 atmosphere, the hydrogen will be absorbed until you reach a vacuum. The understandability of the result and the dynamism of the function are fascinating.

### The relationship of hydrogen absorption and embrittlement is a reversed concept.

That's right. In metals, hydrogen is a problem. If hydrogen concentrates somewhere in a material, the integrity of the metal is weakened and the material may be destroyed at the point, or the material itself can be destroyed by swelling of hydrides. These phenomena, which are generally termed hydrogen embrittlement, are a serious problem in practical applications, so it's desirable that the hydrogen escape from the material as quickly as possible. This in turn is linked to separation/permeability membranes.

After beginning research on separation/permeability membranes, I studied as a foreign student at the Oak Ridge National Laboratory in the United States from 1990 to 1991. My research topic at the time was hydrogen embrittlement of intermetallic compounds. After returning to Japan, I took my degree based on this research. In order to trace the mechanism responsible for embrittlement, it is necessary to determine how much hydrogen is absorbed and how it is diffused. Eventually, I

was able to integrate this work with research on separation/permeability membranes.

### There was a period when the future potential of fuel cells was a focus of attention.

It was a kind of fad. There was a feeling that the fuel cell era was just around the corner, but in reality, things weren't that simple. In 2005, an argument developed to the effect that we needed to go back to the basics and make a careful search for easy-to-use, long-life materials. We launched a project based on this thinking in 2006.

This project consists of four groups. Electrolytes include the high temperature type and the low temperature type. With the former, we are developing materials which can be used at the lower temperature, and with the latter, materials which can be used at higher temperatures. Separators are materials which are inserted between cells when the individual fuel cells are stacked. Conventionally, separators were mainly carbon materials, but because these tend to be expensive, we are using a special high nitrogen stainless steel, which was developed by NIMS. The target of work in Catalyst is to develop a platinum substitute catalyst. This challenge has long been considered a dream, but we have already begun work on it.

I am directly in charge of work on hydrogen purification. High purity hydrogen will be necessary and indispensable in the low temperature-type solid polymer fuel cells which will be used in automobiles in the future. Our objective is to develop a hydrogen separation/permeability membrane that can produce hydrogen of the necessary purity in a single-stage process.

### You are also the Head of the Interdisciplinary Cluster in the Sustainability Field.

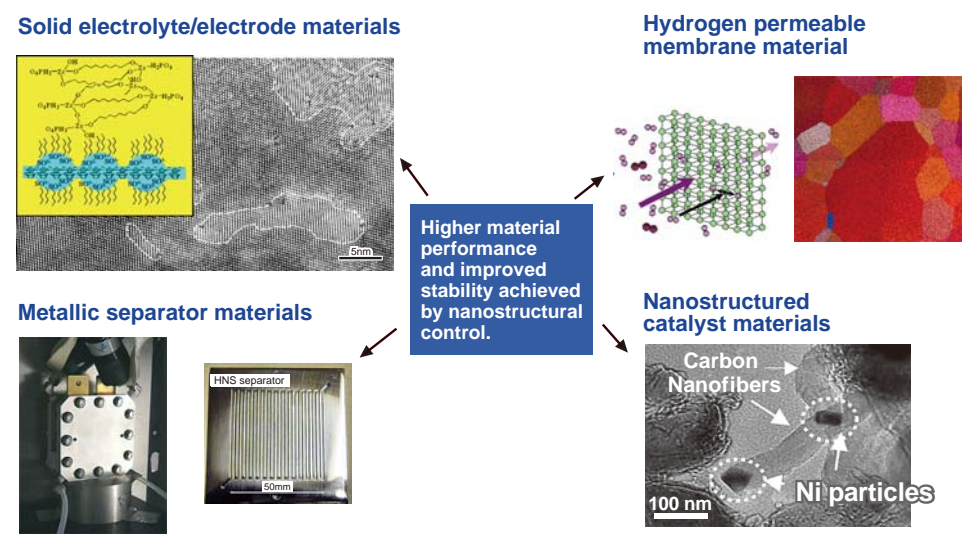
This is a job in which I study research topics and promotion systems for projects under NIMS's next Mid-Term Program, with the aim of developing materials with excellent environmental properties. Although we don't expect to find answers to problems immediately, interdisciplinary topics and basic topics are important.

The separation/permeability membranes that I'm involved with are also an example of this. A separation process is an indispensable process in the production of any materials. However, because the conventional distillation method is energy intensive, separation using a membrane would have a tremendous energy saving effect. To date, separation membranes have centered on organic based materials, and it wasn't really possible for us to grapple head-on with metals and other inorganic materials, but if we researchers in inorganic materials band together to take on the challenge of catalysts and membranes, I think we can produce some very interesting results.

### What are the things that you always keep in mind, and what do you do for a change of pace?

"When you run into a wall, think as widely as possible from various directions." I think that's important. If you don't do that, you can't see where you're going. For an everyday change of pace, I have a drink with my friends and colleagues or play baseball. I enjoy games on Sundays with the baseball club. Recently, I've been a right fielder.

Fig.2



"The value of materials is in their use," which is the basic philosophy of NIMS, expresses the intention to incorporate a variety of material functions in products and systems. For example, NIMS is actively utilizing nanotechnology in the development of fuel cell materials, including solid electrolytes with high conductivity at medium and low temperatures (upper left), high selectivity, high permeability hydrogen permeable membranes (upper right), high efficiency hydrogen production catalysts (lower right), and metallic separator materials with high stability and high conductivity (lower left).



# Materials Data Sheet Station

Aiming at Improved Reliability and Safety in Structural Materials



## Development of a High Accuracy Creep Life Prediction Method

– Aiming at Improved Performance and Improved Safety and Reliability of High Temperature Plants –



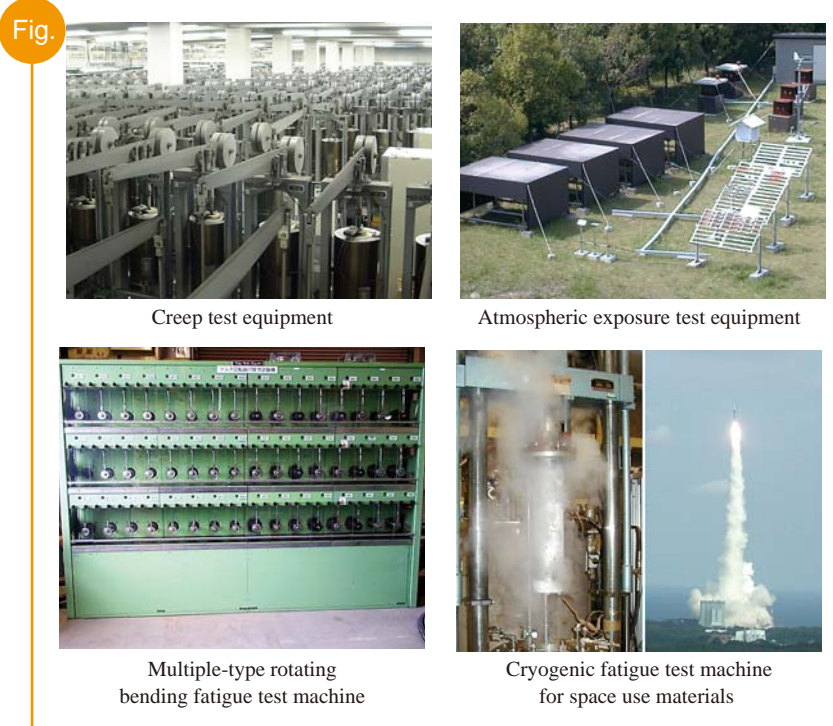
Station Leader  
Kazuhiro Kimura

Accidents due to metal fatigue and similar causes not only threaten safety, but also a serious economic problem. The economic loss associated with accidents due to failure caused by fatigue of metallic materials, and so on, is estimated at approximately 4% of GDP,\*1 while the cost of corrosion countermeasures for metallic materials corresponds to about 1% of GDP.\*2 Combining the two, the annual economic burden is roughly ¥27 trillion (in 2005, Japan's GDP was approximately ¥538 trillion). Reducing this enormous economic loss would make an extremely important contribution to society. Investment in safety, in other words, investment to prevent accidents before they occur, tends to be viewed as "waste" if measures are successful and no accident occurs.\*3 However, providing a common intellectual research infrastructure for research and development to reduce the huge economic loss described above would be a significant contribution to society by science and technology.

On the other hand, a solution to global environmental problems will require a solution to the "trilemma proposition" of (1) economic development of the developing nations, (2) stable supply of electric energy with high use value, and (3) reduction of emissions of CO<sub>2</sub>, which is a greenhouse gas. Because thermal power plants are the largest source of CO<sub>2</sub> emissions in Japan, getting higher efficiency in power generation using higher temperature steam or combustion gas is necessary. One key to an effective solution is the development of high strength heat resistant materials for realizing high efficiency power generation with a lower environmental impact. Because it is impossible to maintain an advanced modern society without a stable supply of electric power, high safety and reliability must be maintained in power plants.

With the aim of improving the reliability of various types of structural materials, which are indispensable for building a safe, secure society, the Materials Data Sheet Station produces and accumulates data on material properties in connection with "Creep," "Fatigue," "Atmospheric Corrosion," and "Strength of Space Use Materials," using the groups of test equipment shown in the accompanying figures, and disseminates this information in the form of structural materials data sheets. Creep is a phenomenon in which time-dependent deformation occurs when external force is applied to an object. Because remarkable creep is observed under high temperatures exceeding 1/2 of the melting point of a material, it is the controlling strength property for the life of high temperature structures. The Materials Data Sheet Station is engaged in the work to elucidate the meaning of the material property data obtained, and in research and development of methods of utilizing this data/knowledge, new test methods and evaluation methods.

For dissemination of results and educational purposes, the Station also holds lectures, seminars, and other events with the aim of contributing to research and development and production activities in the practical world. Its results have been reflected in design standards for thermal and nuclear power plants and Japan's HII-A rocket, among others, and are also widely used in the development of new materials.

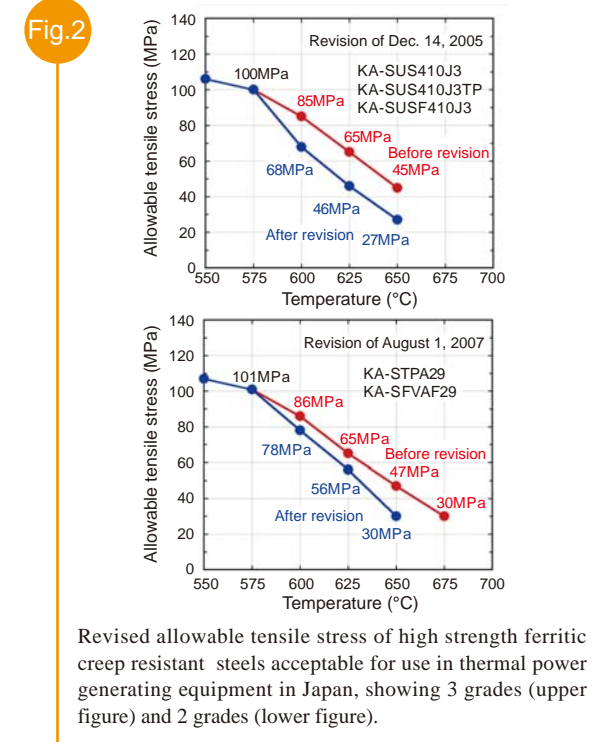
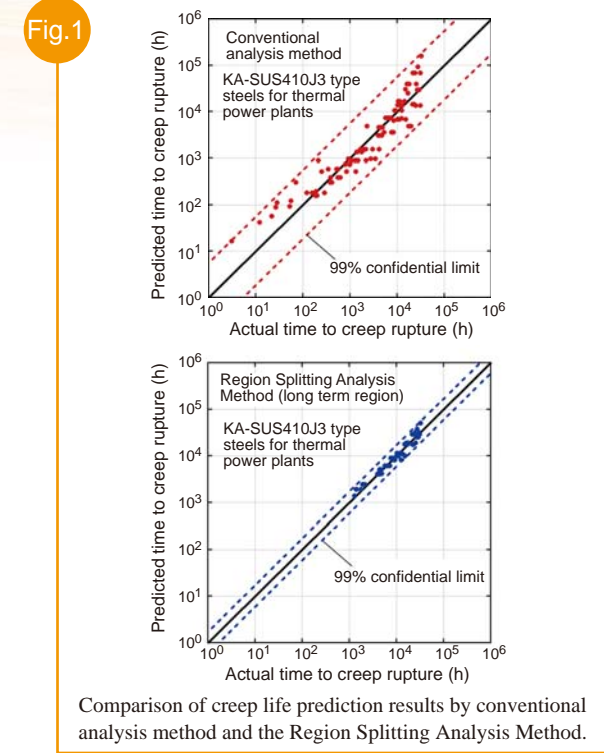


\*1 Economic Effects of Fracture in the United States, Part 1: A Synopsis of the September 30, 1982 Report to NBS by Battelle Columbus Laboratories, National Bureau of Standard, Washington, DC, National Measurement Lab., March 1983, U.S. Department of Commerce, National Information Service.  
\*2 Japan Society of Corrosion Engineering, "Report on Study of Evaluation of Loss due to Corrosion in Japan," May 2001.  
\*3 Yukitaka Murakami, "Frequency of Gigantic Accidents and Modern Science and Technology," NSK Technical Journal, 2003, No. 675, 1-3.

From the viewpoint of conserving limited fossil energy resources and reducing emissions of CO<sub>2</sub> gas, which is considered to be a cause of global warming, higher energy efficiency is demanded in thermal power plants. Higher operating temperatures are necessary in order to improve energy efficiency, but adoption of higher temperatures creates a more severe service environment for materials and in particular, accelerates time-related deterioration of structural materials. Boilers, pressure vessels, and other equipment are designed and manufactured based on allowable stress. The allowable stress in high temperature is generally determined by the stress at which creep rupture occurs in 100,000 hours (10<sup>5</sup> hours; approx. 11 years and 5 months). Accordingly, in order to secure the safety of high temperature equipment, it is important to evaluate long-term, 10<sup>5</sup> hour creep strength with high accuracy.

The high strength ferritic creep resistant steels developed in recent years have made it possible to increase the steam temperature in thermal power plants from the conventional 566°C to around 600°C, and thus have made a substantial contribution to improving energy efficiency. However, with these high strength ferritic creep resistant steels, over-estimation of the long-term creep strength of the material had been a concern. Therefore, NIMS researchers investigated the mechanism of strength reduction accompanying long-term use at high temperature, and carried out a study aimed at achieving high accuracy in a long-term creep life prediction method based on the strength reduction mechanism. This work led to the proposal of the "Region Splitting Analysis Method." This "Region Splitting Analysis Method" is a technique in which creep life is analyzed independently in the high stress regime and low stress regime, in which different factors control creep deformation. As shown in Fig. 1, long-term creep life can be predicted and evaluated with higher accuracy with the region splitting analysis method than with the conventional method, in which all data are analyzed collectively.

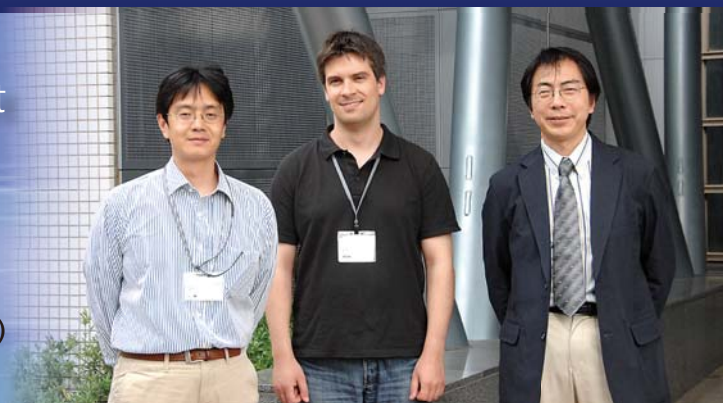
In Japan, the "Region Splitting Analysis Method" was adopted in a reevaluation of the long-term creep strength of high strength ferritic creep resistant steels. Based on the evaluation results, the allowable tensile stresses of 5 steel grades were revised in December 2005, and an additional 7 grades were revised in August 2007. An example of these revisions is shown in Fig. 2. Evaluation results by the "Region Splitting Analysis Method" were also adopted in the remaining life evaluation equations for high temperature equipment in existing plants. Thus, this method will contribute to improvement of safety and reliability, not only in high temperature equipment designed and manufactured in the future, but also in high temperature equipment which is already in service. Our policy is to promote testing and research on long-term creep strength characteristics with the aim of securing improved performance and higher safety and reliability in high temperature equipment in the future.





## Fabrication of Metallic Nanosheet for High Sensitivity Plasmon Enhanced Sensor

International Center for Materials Nanoarchitectonics (MANA)  
Nano System Functionality Center<sup>†</sup>



Tadaaki Nagao, Dominik Enders, Tomonobu Nakayama<sup>†</sup>  
Managing Director

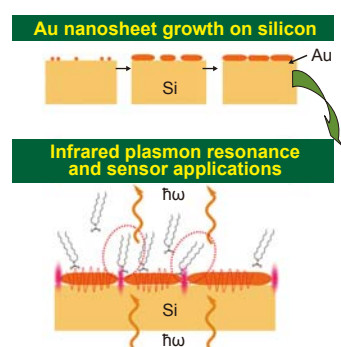
Oscillation of free electrons in metals is termed plasmon. In vibrational spectroscopy, it is known that the signal intensity of the oscillation of molecules adsorbed on the surfaces of metallic nanoparticles and nanofilms can be enormously increased, resulting in enhanced detection sensitivity. Using this phenomenon, noncontact/nondestructive chemical and biosensing techniques become widespread. Among these techniques, surface enhanced infrared absorption spectroscopy has attracted particular attention in recent years for its simplicity and high sensitivity, and is being applied to the monitoring of chemical reaction and micro-analysis of catalytic surfaces and electrode surfaces.

In order to realize the high sensitivity, it is necessary to optimize the following two conditions: (1) reduction of the spacing between the nanostructures and to enhance the electrical field convergence there, and (2) flattening of the individual nanostructures and integration of those nanostructures. Although the signal sensitivity is strongly governed by the structure of the metallic nanomaterial used, it is difficult to fabricate precisely the nanostructure with high reproducibility, and thus the sensitivity varies largely each time it is fabricated. In order to overcome this difficulty, we have developed a technique for producing a flat metallic nanosheet at the silicon/solution interface (Fig. 1, top), using spherical gold nanoparticles as the starting material and monitored its growth *in situ*. For monitoring the growth, we used the attenuated total reflection (ATR) method, which has high sensitivity for solid-liquid interfaces, and measured the

water band in the solution in the infrared absorption spectrum (Fig. 2, lower left). As the metallic sheets grew, the gap between the sheets decreased, and the flatness of the nanosheets also increased. Broad plasmon resonance occurred in the infrared band as a result of this structural change in the nanosheets. This plasma oscillation and the oscillation of the water molecules interacted, resulting in a change in the infrared absorption spectrum. We found that it is possible to fabricate optimized structures with good reproducibility by monitoring this spectrum profile and stopping the film growth just before the films percolates (Fig. 2, upper left). When thiol molecules were adsorbed on the fabricated sensor film and measured by the IR beam, a gigantic absorption intensity exceeding 20% could be obtained even with a single layer of adsorbed molecules (Fig. 2, right). This signal intensity is more than 15 times stronger than that of spherical nanoparticles.

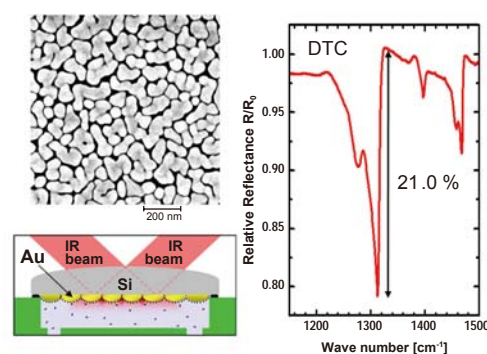
Once the growth condition of the optimized sensor films is established, the fabrication method is also applicable to ordinary silicon wafers, glass substrates, and similar materials. Development to a more practical sensing technique is also expected. In the future, we plan to develop practical materials with higher sensitivity, while exploring various materials and fabrication conditions with this method.

Fig. 1



(Top) Schematic diagram of 2-dimensional grown of a gold nanosheet on silicon.  
(Bottom) Example of measurement by infrared absorption spectroscopy (transmission method). A large infrared absorption signal occurs when molecules enter a nano gap where the electrical field is concentrated.

Fig. 2



(Upper left) Scanning electron microscope image of a gold nanosheet film with optimized sensitivity.  
(Lower left) Schematic illustration of ATR measurement, showing detection of a trace amount of ocatadecanethiol (ODT), dithiolcarbamate (DTC), or a similar substance in solution, which is adsorbed as a single molecular layer on a gold nanosheet on a silicon substrate (gray).  
(Right) Infrared absorption spectrum of a single molecular layer of DTC. A gigantic absorption signal as large as 21% is produced.

## Superhydrophobic Films Based on Fullerene Materials

Macromolecules Group, Organic Nanomaterials Center  
Max-Planck Institute of Colloids and Interfaces (Germany)



Takashi Nakanishi

The C<sub>60</sub> fullerene, which consists of 60 carbon atoms, is a promising nano carbon material, with expected applications including electronic and medical materials, hard additives in polymers, etc. However, since their implementation in practical applications is still limited, the development of new material applications is desired. The self-cleaning feature shown by the lotus leaf in the natural world is based on a superhydrophobic (super-water repellent) property by which the leaf surface repels water. Active developments of artificial (mimetic) superhydrophobic films are underway using fluorine-based polymers, among other substances. However, even though it is known that fullerenes have small surface free energy, one of requirements for superhydrophobic materials, superhydrophobic materials based on fullerenes have not been explored.

In a compound (Fig. a) in which long chain alkyls are introduced in C<sub>60</sub> fullerene as a substituent, spherical microparticles (diameter: several μm) with a nanometer-sized flake structure on the outer surface (Fig. c) are formed by performing a simple operation of heating and cooling in a 1,4-dioxane solvent. In the structural subunit (molecularly-assembled) structure which forms this microparticle, the alkyl component has an interdigitated arrangement (alternating overlapping structure), while the fullerene component has a two-dimensional layer structures (Fig. b). A thin film with a surface having microscopic (nano/micro order) irregularities can be produced simply by coating a dispersed solution of

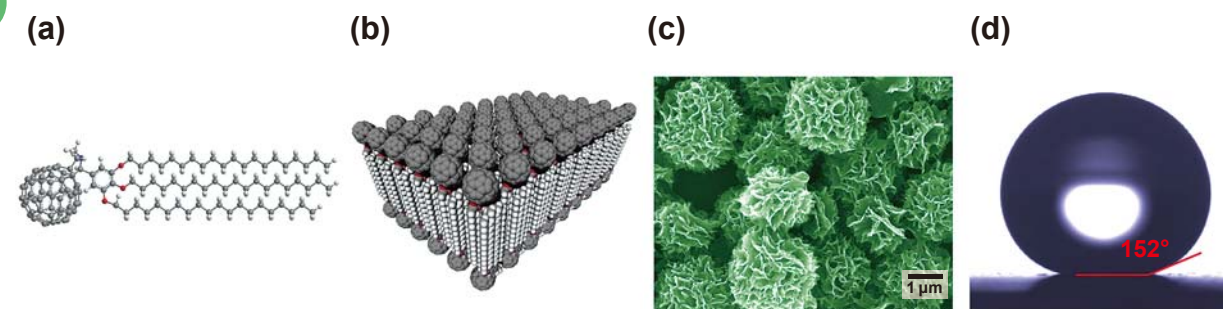
these microparticles on a substrate and allowing the coating to dry. It was found that the contact angle of water at this surface displays superhydrophobicity, having an angle of 152° (Fig. d). This water-repellant film not only shows durability towards polar organic solvents such as acetone and ethanol, but also has excellent solvent durability to acidic and basic aqueous solutions. Furthermore, the film also possesses thermal stability, absolutely no effect on its water-repellant property was observed when the film was exposed for more than 36 hours at 100°C. These properties are achieved because the π-π interaction between the fullerenes is extremely strong, even in polar solvents and under heating environments.

In addition to the simplicity of the facile fabrication method, because the film is readily dissolved in good solvents for fullerene compounds (chloroform, toluene), recovery and reuse of the compound are surely possible, and consideration can also be given to sustainable chemistry. Thus, this film can be a versatile surface coating material for applications requiring superhydrophobicity.

A part of this research was supported by the Japan Science and Technology Agency's PRESTO (Precursory Research for Embryonic Science and Technology) program, "Structure Control and Function."

T. Nakanishi, et al., *Adv. Mater.* 2008, 20, 443-446.

Fig.



(a) Fullerene derivative bearing long alkyl chains used in this research  
(b) Structural subunit structure self-organized of the fullerene derivatives.  
(c) Scanning electron microscope image of microparticles with nano-flake structure on the outer surface.  
(d) Photograph of the contact angle of a water droplet on the microparticle thin film.



## Introduction of New Senior Distinguished Adviser

### Comments on Appointment as NIMS Senior Distinguished Adviser

I was recently appointed as a Senior Distinguished Adviser and Head of the newly established Graduate School Team as well. NIMS has already signed agreements and begun operation of the Joint Graduate School Program for several years with many of Japan's leading universities. In this Program, NIMS researchers have been providing research guidance to graduate students as Visiting Professors and Associate Professors. In 2004, NIMS established a new type of Joint Doctoral Program called the "Doctoral Program in Materials Science and Engineering" in the University of Tsukuba. In this Program, NIMS itself selects and accepts outstanding students from Japan and overseas, and then provides research guidance at NIMS. As is well known, the United States and many leading countries in Europe are fiercely competing in collecting promising young researchers from around the world to activate and implement their own country's scientific and technological development. The NIMS Graduate School Team is focusing its efforts on expanding the Tsukuba-type "Joint Doctoral Program" to other universities to collect promising graduate students from overseas in wider areas of research and vitalize NIMS. In order to understand our current situation, we have finished personal interviews with more than 100 graduate students currently doing research in NIMS. In this new post, I hope to take advantage of my experience to date to increase the level of activity and internationalization at NIMS.

### Kenichi Shida



Graduated from the Department of Applied Physics, Waseda University (BA, 1963). Completed Master Course of the Graduate School of Engineering (Applied Physics), The University of Tokyo (ME, 1965). Joined Kurashiki Rayon Co., Ltd. (now Kuraray Co., Ltd., 1965) as a researcher in the Central Research Laboratory, and subsequently worked in the Head Office. Appointed Vice President of Kuraray International Corporation (New York, 1984), and later returned to the Kuraray Head Office (1988). Named Managing Director and Secretary General of The Society of Polymer Science, Japan (1996) and Representative of the Tokyo Office of Hokkaido University (2004). He was appointed Senior Distinguished Adviser, National Institute for Materials Science in 2008.

## KIER (Korea) and NIMS Held First International Joint Workshop

(Mar. 13-15, 2008) – An international joint workshop was held at the Korea Institute of Energy Research (KIER) in Deajon City, Korea over a period of 3 days and 2 nights by the NIMS Nano Ceramics Center and KIER's Future Technology Research Division. The joint workshop was planned in order to strengthen substantial research exchanges with KIER, with which NIMS concluded an MOU in the field of ceramics in December, 2007. Seven researchers from NIMS visited Korea for this event, including Dr. Yoshio Sakka, the Managing Director of the Nano Ceramics Center. The first day featured a tour of KIER's facilities and banquet hosted by KIER. On the second day, a total of 14 presentations were given by the Korean and Japanese sides, with approximately 80 researchers participating. In addition to active debate on advanced ceramics research at the respective institutes, close research cooperation in the future was also discussed. After the seminar, the participants also toured ceramics-related laboratories. In the morning before their return to Japan, the Japanese researchers visited cultural sites in Daejon City, guided by researchers from KIER, thereby deepening their friendship with researchers at the collaborating institute on the Korean side while also exploring Japanese-Korean history. In particular, scenes of this visit to Korea by NIMS and the workshop were broadcasted by the Daejon TV station, providing a good opportunity to increase knowledge of NIMS. In the future, Japan-Korea workshops will be held periodically. The next such event is scheduled for next year in Japan.

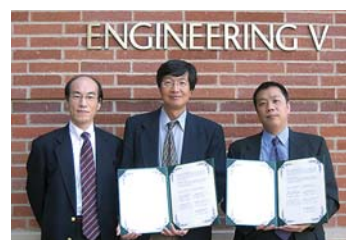


Presenters participating in the workshop

## Deepening of International Cooperation between UCLA and NIMS

### In March 2008 two memorandums of understanding (MOU) were signed with University of California, Los Angeles (UCLA)

(Mar. 24, 2008) – NIMS President Kishi met with UCLA Chancellor Block in Tokyo. Prof. Kishi handed over a letter of support for the proposed Center for the Environmental Impact of Nanotechnology (CEIN) at UCLA, which addresses the potential risk of the use of nanotechnology. Then Prof. Kishi and Dr. Block signed the MOU between the International Center for Materials Nanoarchitectonics (MANA) at NIMS and the California NanoSystems Institute (CNSI) at UCLA for the opening of the first MANA satellite at UCLA in April 2008.



From left: Prof. Kagawa, Group Leader, NIMS Prof. J-M. Yang, UCLA and Dr. Naito, NIMS.

(Mar. 20, 2008) – The NIMS Composites and Coating Center signed a MOU for collaboration on "Developing high performance carbon fibers and hybrid composites" with the Department of Materials Science and Engineering at UCLA.



NIMS President Prof. Kishi (left) and UCLA Chancellor Dr. Block

## Newly Established "NIMS-Hokkaido University Joint Doctoral Program" in Two Fields

On May 1, 2008 a Joint Doctoral Program in the field "Advanced Functional Chemistry" was established in the Hokkaido University Graduate School of Division of Chemistry, Japan. This was followed on June 1 by the establishment of a new Joint Doctoral Program in the field of "Frontier Biomaterials Science" in the Graduate School of Life Science, Biosystems Science Course. In both fields, NIMS will serve as the parent body, and NIMS researchers will have the primary responsibility for providing guidance to students in the role of instructors. In distinction from the conventional "Joint Graduate School System," NIMS calls this new system the "Joint Doctoral Program System."

The "Advanced Functional Chemistry" field will be operated as a joint project of Hokkaido University's Global COE "Catalysis as the Basis for Innovation in Materials Science" and the NIMS International Center for Materials Nanoarchitectonics (MANA). NIMS researchers will provide research guidance to doctoral course students as adjunct professors or adjunct associate professors in the highest-level educational and research environment in Japan.

The "Frontier Biomaterials Science" field will pioneer a new interdisciplinary field by a fusion of life science and materials/nanotechnology. With the aim of creating innovative technologies, education to enable students to acquire both knowledge and experience through research activities will be provided under the guidance of NIMS researchers as academic advisers, and members in the Hokkaido University Graduate School of Life Science as assistant academic advisers.

In both fields, the aim is to train young human resources who will truly play an active role in international society by providing more effective educational and research guidance, taking advantage of the outstanding research environment at NIMS.

This new joint program is the second for NIMS, following the establishment of the Doctoral Program in Materials Science and Engineering with the University of Tsukuba in 2004. Nationwide expansion of this new Joint Doctoral Program is planned, based on a careful consideration of the special characteristics of counterpart universities and their regions.

## The NIMS Annual Open House

(Apr. 18-20, 2008) The NIMS Annual Open House event was held April 18-20, 2008 as part of Japan's Science and Technology Week. More than 40 research institutes and laboratories in the Tsukuba area are open to the public during this week every year.

NIMS offered 54 individual programs for the enjoyment of visitors, including a neodymium magnet craft class, dancing coins in a high magnetic field, a human-size bubble, facility tours, and of course, there were innovative programs for people particularly interested in materials science. At the Special Children's Events on April 20, many children were excited to make various kinds of crafts by themselves.

A total of 1644 visitors came to NIMS Open House Events. NIMS sincerely hopes that these experiences will help all visitors understand and connect with NIMS and enjoy what is truly fun in science, and will encourage young students to become great scientists in the future.

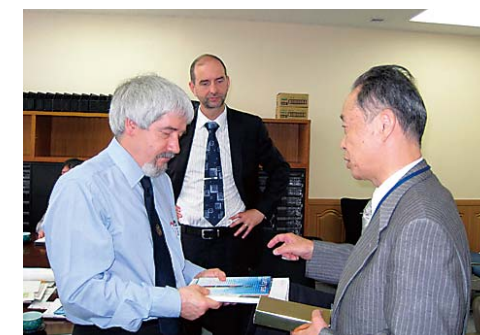


Demonstration of iron casting

## Professor David Delpy, Chief Executive of EPSRC visits NIMS

(May 23, 2008) Professor David Delpy, Chief Executive of Engineering and Physical Sciences Research Council (EPSRC) and Dr. Paul Johnson, First Secretary of British Embassy to Japan visited NIMS. They met Professor Yukichi Umakoshi, the Vice President of NIMS, Professor Sukekatsu Ushioda, the NIMS Fellow and Dr. Masaki Kitagawa, the Senior Distinguished Advisor of NIMS, Dr. Kotobu Nagai, the Coordinating Director, and Dr. Chikashi Nishimura, the Managing Director of Fuel Cell Center.

They exchanged information on research trends and support and large scale infrastructure construction. In the lab. tour, Prof. Delpy visited 3D Nano-Integration line and the Rolls-Royce Centre of Excellence for Aerospace Materials.



From left: Prof. D. Delpy, Dr. P. Johnson and Prof. Y. Umakoshi

## Doctoral Program in Materials Science and Engineering Graduate School of Pure and Applied Sciences, University of Tsukuba

### Entrance Exam for April 2009 Admission

The Doctoral Program in Materials Science and Engineering will be held an Entrance Exam in August 2008 for enrollment in April 2009. The program is jointly managed by NIMS and the University of Tsukuba, and the scientists selected from NIMS have joined the graduate school faculty and supervised students' thesis research.

Please feel free to contact us for details at anytime.

#### SCHEDULE

Distribution of Application Form	The form is available now
Application Period	July 22-24, 2008
Oral Examination*	August 19-20, 2008
Announcement of the Results	September 2, 2008
Enrollment	April 1, 2009

\*At oral examination, applicants must submit the Score card of English proficiency test (TOEIC or TOEFL).

#### CONTACT

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For more details: <http://www.nims.go.jp/graduate/english/>



A scene at the seminar



## Hello from NIMS

### Overseas Experience in Tsukuba



Hello, I am Yang Huang. After finishing my master program in China, I came to Japan last April for my Ph.D. studies. I am participating in the joint doctoral program of NIMS and the University of Tsukuba. On one hand, I am a real student in the campus. There I can take Japanese classes and experience the youthful spirit and culture difference. I enjoyed the "Sohosai," an annual festival at the University of Tsukuba, and like the Hokkaido folk song "Soran Bushi" very much.

At NIMS, I mainly focus on scientific research. My research theme is BN-based one-dimensional nanomaterials and is supervised by Prof. Yoshio Bando and Prof. Dmitri Golberg. I feel very lucky as a member of such a world-famous group, which is pioneering studies on inorganic nanotubes, and expect to learn a lot in the future from my mentors. This expectation seems to be not unreachable when I discuss my findings with the world-renowned professors, when I use the state-of-the-art synthesis and

microscopy facilities, and when I enjoy the enlightening seminars given by the outstanding researchers.

Out of the campus and the Institute, I fully enjoy my daily life. The environment in the Tsukuba Science City is most attractive. The numerous parks, trees, flowers and even farmland make my heart and soul peaceful. Owing to the fact that many foreign researchers reside here, different cultures come together in Tsukuba as in "a melting pot." It is possible to try foods with different tastes. I can easily find here diverse restaurants characteristic of Japan, China, Italy, Iran, Korea, Thailand, and many other countries.

One year has passed since I came to Japan with the common worries about how to adapt myself to the new life. Now I have become much more confident of my future and am starting to entirely enjoy my overseas life.

Yang Huang (China)  
University of Tsukuba, Ph.D. Student/NIMS  
Junior Researcher (April 2007 - Present)  
Nanomaterials Synthesis & Analysis Group,  
Nanoscale Materials Center



[ Oversea life starts from here. ]



[ Enjoying "Hanabi" (a firework festival) with my friends from NIMS and the University of Tsukuba. ]



National Institute for Materials Science

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